

PATENT SPECIFICATION

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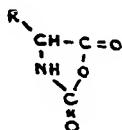
International Classification:—C08g.

COMPLETE SPECIFICATION

Improvements in and relating to the Production of Polyamides

We, COURTAULDS LIMITED, a British Company, of 16, St. Martin's-le-Grand, in the City of London, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to the production of polyamides and particularly to the production of synthetic polypeptides by polymerising anhydrocarboxyamino-acids having the general formula:

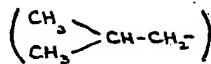


R being an alkyl or aralkyl group, which may be substituted with an inactive substituent group such as an ester group. Examples of suitable anhydrocarboxyamino-acids are as follows, the R group being indicated in brackets:

The anhydrocarboxyamino-acid of gamma-benzyl-L-glutamate

($\text{C}_6\text{H}_5-\text{CH}_2-\text{O}-\text{OC}-\text{CH}_2-\text{CH}_2-$), of DL-beta-phenyl-alanine ($\text{C}_6\text{H}_5-\text{CH}_2-$),

of gamma-methyl-L-glutamate ($\text{CH}_3-\text{O}-\text{OC}-\text{CH}_2-\text{CH}_2-$), of leucine



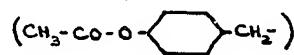
of isoleucine ($\text{CH}_3-\text{CH}_2-\text{CH}-$), of



norleucine ($\text{CH}_3-\text{CH}_2-\text{CH}_2-\text{CH}_2-$), of epsilon-N-carboxy-benzoxy-lysine ($\text{C}_6\text{H}_5-\text{CH}_2-\text{O}-\text{OC}-\text{NH}-\text{CH}_2-\text{CH}_2-$

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CH_2-CH_2-), of ortho-acetyl tyrosine



and also alpha-amino-N-butyric-anhydro-carboxy amino acid ($\text{CH}_3-\text{CH}_2-\text{CH}_2-$).

The term "polypeptide" as used in this specification means a polyamide built up with a recurrent



group and obtained by polymerising an anhydrocarboxyamino-acid having the formula defined above.

It is known that anhydrocarboxyamino-acids can be polymerised by heating, either alone or in the presence of certain catalysts, the polymerisation being accompanied by the evolution of carbon dioxide. The catalysts generally proposed hitherto are water, tertiary bases such as pyridine, and compounds of the type XH in which H is an active hydrogen atom. Examples of such compounds are primary amines, constant at 25°C . in the range of 2.25×10^{-3} to 6×10^{-1} .

The object of this invention is to promote rapid polymerisation of anhydrocarboxyamino-acids.

In accordance with the present invention the polymerisation of one or more anhydrocarboxyamino-acids is effected in solution in the presence of penta-methyl guanidine. The penta-methyl guanidine is preferably added to the anhydrocarboxyamino-acid as a solution for example in N-methyl formamide.

The polymerisation of the anhydrocarboxyamino-acid is effected in solution. Suitable solvents are benzene, nitrobenzene, chlorobenzene, acetophenone, dioxane, methylene

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Price 4s 6d.

Price 25p

chloride, chloroform, dimethyl formamide and mixtures of these compounds, the actual choice of solvent depending on the solubility properties of the original anhydrocarboxy-amino-acid or acids and the resulting polypeptide. The polymerisation can be carried out at varying temperatures; temperatures ranging from 0° to 100° C. have been used successfully. For economic reasons it is preferred to use ordinary temperatures.

The invention is illustrated by the following Example in which parts are by weight.

EXAMPLE.

5 parts of the N-carbonic anhydride of gamma-benzyl-L-glutamate were dissolved in 100 parts of dioxane. A solution of 0.032 part of pentamethyl guanidine (1.32 mol per cent. based on the weight of the anhydride) in 1 part of N-methyl formamide was added with stirring. The resultant solution was stirred for 15 minutes at 25° C. in an evacuated container while a rapid evolution of carbon dioxide occurred. The polymer was precipitated by the addition of 2000 parts of ether.

The product obtained was a colourless fibre-forming polymer; its reduced viscosity in di-

chloracetic acid was 0.56. Reduced viscosity

is given by $\frac{\eta_{sp}}{c}$ where η_{sp} is the specific viscosity of a polymer solution having a concentration (c) of 0.5 gram per 100 cc. of solvent.

The procedure described in the Example may be used for polymerising other anhydrocarboxyamino acids such as the N-carbonic anhydride of DL-β-phenylalanine.

What we claim is:—

1. A process for the production of polyamides which comprises polymerising one or more anhydrocarboxyamino acids as hereinbefore defined in solution in the presence of penta-methyl guanidine.

2. A process for the production of polyamides as claimed in claim 1 carried out substantially as described in the foregoing Example.

3. Polyamides which have been produced by the process claimed in either of the preceding claims.

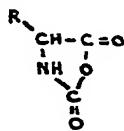
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PROVISIONAL SPECIFICATION

Improvements in and relating to the Production of Polyamides

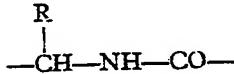
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It is known that anhydrocarboxyamino-acids can be polymerised by heating, either

alone or in the presence of certain catalysts, the polymerisation being accompanied by the evolution of carbon dioxide. The catalysts generally proposed hitherto are water, tertiary bases such as pyridine, and compounds of the type XH in which H is an active hydrogen atom. Examples of such compounds are primary amines, secondary amines, phenols, and acids having an ionization constant at 25° C. in the range 2.25×10^{-3} to 6×10^{-7} .

The object of this invention is to promote rapid polymerisation of anhydrocarboxyamino-acids.

In accordance with the present invention the polymerisation of anhydrocarboxyamino-acids is effected in the presence of a penta-alkyl guanidine, for example penta-methyl guanidine. The substituted guanidine is preferably added to the anhydrocarboxyamino-acid as a solution for example in N-methyl formamide.

The polymerisation of the anhydrocarboxyamino-acid is effected in solution. Examples of suitable solvents are benzene, nitrobenzene, chlorobenzene, acetophenone, dioxane, methylene chloride, chloroform, dimethyl formamide and mixtures of these compounds, the actual choice of solvent depending on the solubility properties of the original anhydrocarboxyamino-acid or acids and the resulting polypeptide. The polymerisation can be carried out at varying tem-

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